Fiber (Crude) in Animal Feed: Fritted Glass Crucible Method

Scope

Crude fiber is the loss on ignition of dried residue remaining after the digestion of the sample. The method is applicable to grains, meals, flours, feeds and fiber bearing material from which fat can be extracted to leave a workable residue.

Summary

The fat is extracted from the sample and the sample digested with sulfuric acid and sodium hydroxide. The digestion residue is heated in a muffle furnace to ignite the fiber material.

Comments

All of the sample solutions are heated to prevent gelling or precipitation of possibly saturated solutions.

Apparatus and Materials

A. Alundum boiling chips.

B. Fiber beaker, 600 ml.

C. Fritted glass crucible, 50 ml, coarse porosity.

D. Desiccator.

E. California buchner funnel.

F. Continuous solution heater.

G. Fiber digestion apparatus.

H. Muffle furnace.

I. Oven, 130°C.

Reagents

A. Sulfuric Acid Solution 1.25% (0.255N): Prepare by diluting 142 ml of concentrated sulfuric acid to 20 liters with deionized water. The concentration must be checked by titration against standardized 0.25N sodium hydroxide.

B. Sodium hydroxide solution 1.25% (0.313N): Prepare by diluting 298 ml of 50% solution (18.9 N) to 18 liters with deionized water. The concentration must be checked by titration against standardized 0.25N sulfuric acid.

Procedure

A. Extract the fat from a weighed sample (approximately 2.0 g weighed to the nearest 0.0001 g) using the usual direct anhydrous ether method.

B. After the ether has been removed from the sample, transfer the sample to a 600 ml fiber beaker containing three alundum boiling chips.

C. Add 200 ml of boiling 0.255 N sulfuric acid and place on a preheated digestion apparatus.

D. Boil for 30 minutes.

E. Filter the sample through a California buchner funnel with suction and wash the residue four times with boiling water (40-50 ml per wash). Do not add wash to the funnel under vacuum; lift the funnel from the apparatus when adding wash.

F. Wash the residue from the funnel back into the 600 ml beaker with boiling 1.25% sodium hydroxide.

G. Add a total of 200 ml of 1.25% sodium hydroxide.

H. Place the beaker on a pre-heated burner or the digestion apparatus and boil for 30 minutes.

I. Decant the liquid through a heated, fritted glass crucible and wash the solids into

the crucible with a minimum of near boiling deionized water.

J. Wash the residue once with near boiling sulfuric acid solution (approximately 25-30 ml).

K. Wash the residue twice with boiling deionized water (approximately 25-30 ml each time), filtering after each washing.

L. Dry the crucible with residue for 2 hr at 130°C or at 100°C overnight.

M. Remove the sample from the oven and cool in the desiccator.

N. Weigh the residue and crucible to the nearest 0.0001 g.

O. Place the residue and crucible in a cool muffle furnace and set it to 550°C. Turn on the furnace.

P. Ash the sample for two hours at 550 ± 10^{o}. Turn off the furnace after two hours.

Q. Allow the muffle furnace to cool down to $\leq 250^{o}$.

R. Remove the crucibles from the muffle furnace and cool them in a desiccator.

S. Weigh the sample residue and crucible to the nearest 0.0001 g.

Calculations

A. Calculate the loss in weight on ignition by subtracting the weight of the residue and crucible after ignition from the weight of the residue and crucible before ignition.

$$\%\text{Crude fiber} = \frac{(\text{loss in weight on ignition})(100)}{\text{sample weight in grams}}$$

Quality Control

A. Concentrations of sulfuric acid and sodium hydroxide solutions.

1. Check by standardization after preparation adjust if neccesary, and

document.

2. Re-check at three month intervals and document.

B. Temperature of drying oven and ashing furnace.

1. Check the temperature of the drying oven before use with a calibrated thermometer and document on worklist.

2. Check the temperature of the ashing furnace every three months and document.

C. Monitor time.

1. Digestion times in acid and base solutions should be exactly 30 minutes.

2. Crucibles and samples should be dried two hours at 130^{o} or overnight at 110^{o} after filtration.

3. Crucibles and samples should be ashed for two hours at 550 ± 10^{o} after drying.

Bibliography

Official Methods of Analysis (1984) 14th Ed., AOAC, Washington, D.C., secs. 7.071-7.073